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# Sertifikaat / Certificate

ATENTKANTOOR REPUBLIC OF SOUTH AFRICA — REPUBLIEK VAN SUID-AFRIKA PATENT OFFICE

EPARTEMENT VAN HANDEL
N NYWERHEID

DEPARTMENT OF TRADE AND INDUSTRY

Hiermee word gesertifiseer dat
This is to certify that

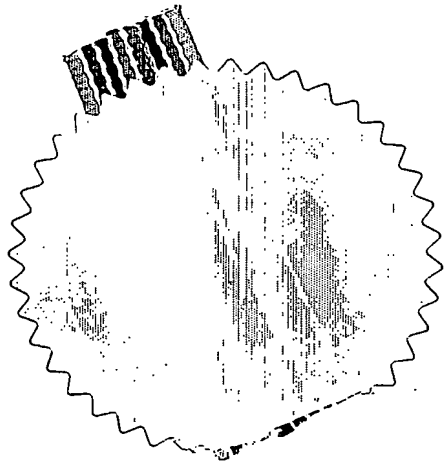


the documents attached hereto are true copies of the Forms P2, P6, provisional specification and drawings of South African Patent Application No. 2002/5993 in the name of Sasol Wax (SA) (PTY) LTD

Filed : 26 July 2002

Entitled : Fibres Containing an Active Ingredient

Geteken te PRETORIA in die Republiek van Suid-Afrika, hierdie 19th dag van August 2003
Signed at in the Republic of South Africa, this day of



Registrateur van Patente

**PRIORITY DOCUMENT**

SUBMITTED OR TRANSMITTED IN COMPLIANCE WITH RULE 17.1(a) OR (b)

FORM P.2

| REPUBLIC OF SOUTH AFRICA | REGISTER OF PATENTS | PATENTS ACT, 1978 |
|---|---|---|

| OFFICIAL APPLICATION | LODGING DATE: PROVISIONAL | ACCEPTANCE DATE |
|---|---|---|
| 21 01 2002/5993 | 22 26 JUL 2002 | 47 |
| **INTERNATIONAL CLASSIFICATION** | **LODGING DATE: COMPLETE** | **GRANTED DATE** |
| 51 | 23 | |

**FULL NAME(S) OF APPLICANT(S)/PATENTEE(S)**

71 ~~SCHUMANN SASOL SOUTH AFRICA (PROPRIETARY) LIMITED~~ SASOL WAX (S.A)(PTY) LTD

NAME CHANGED TO 03.03.03

| APPLICANTS SUBSTITUTED: | DATE REGISTERED |
|---|---|
| 71 | |

| ASSIGNEE(S) | DATE REGISTERED |
|---|---|
| 71 | |

**FULL NAME(S) OF INVENTOR(S)**

72
1. BENETTI, ALDO
2. FOURIE, JOHAN
3. ZWANE, IVOR MZWANDILE

| PRIORITY CLAIMED | COUNTRY | NUMBER | DATE |
|---|---|---|---|
| N.B. Use International abbreviation for country (see Schedule 4) | 33 NIL | 31 NIL | 32 NIL |

**TITLE OF INVENTION**

54 FIBRES CONTAINING AN ACTIVE INGREDIENT

**ADDRESS OF APPLICANT(S)/PATENTEE(S)**

1 KLASIE HAVENGA ROAD, SASOLBURG, 1947, FREE STATE, SOUTH AFRICA

| ADDRESS FOR SERVICE | S & F REF |
|---|---|
| 74 SPOOR & FISHER, SANDTON | PA132143/P |

| PATENT OF ADDITION NO. | DATE OF ANY CHANGE |
|---|---|
| 61 | |
| **FRESH APPLICATION BASED ON** | **DATE OF ANY CHANGE** |
| | |

SPOOR & FISHER

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978

# APPLICATION FOR A PATENT

AND ACKNOWLEDGEMENT OF RECEIPT
(Section 30 (1) – Regulation 22)

REPUBLIC OF SOUTH AFRICA FORM P.1
REVENUE
26.07.02
R 0060.00
HASR 711
INKOMSTE
REPUBLIEK VAN SUID AFRIKA

The granting of a patent is hereby requested by the undermentioned applicant on the basis of the present application filed in duplicate

| OFFICIAL APPLICATION NO. | | S & F REFERENCE |
|---|---|---|
| 21 | 01 2002/5993 | PA132143/P |

(10.03 CHANGED TO 10.03)

FULL NAME(S) OF APPLICANT(S)

| | |
|---|---|
| 71 | ~~SCHUMANN SASOL SOUTH AFRICA (PROPRIETARY) LIMITED~~ SASOL WAX (S.A) (PTY) LTD |

ADDRESS(ES) OF APPLICANT(S)

| | |
|---|---|
| | 1 KLASIE HAVENGA ROAD, SASOLBURG, 1947, FREE STATE, SOUTH AFRICA |

TITLE OF INVENTION

| | |
|---|---|
| 54 | FIBRES CONTAINING AN ACTIVE INGREDIENT |

THE APPLICANT CLAIMS PRIORITY AS SET OUT ON THE ACCOMPANYING FORM P.2. THE EARLIEST PRIORITY CLAIM IS:

| COUNTRY: | NIL | NUMBER: | NIL | DATE: | NIL |
|---|---|---|---|---|---|

~~THIS APPLICATION IS FOR A PATENT OF ADDITION TO PATENT APPLICATION NO.~~

| | | |
|---|---|---|
| 21 | 01 | |

~~THIS APPLICATION IS A FRESH APPLICATION IN TERMS OF SECTION 37 AND IS BASED ON APPLICATION NO.~~

| | | |
|---|---|---|
| 21 | 01 | |

THIS APPLICATION IS ACCOMPANIED BY:

- ☒ 1. A single copy of a provisional specification of 6 pages.
- ☐ 2. Drawings of sheets
- ☐ 3. Publication particulars and abstract (Form P.8 in duplicate).
- ☐ 4. A copy of Figure of the drawings (if any) for the abstract.
- ☐ 5. Assignment of invention.
- ☐ 6. Certified priority document.
- ☐ 7. Translation of the priority document.
- ☐ 8. Assignment of priority rights.
- ☐ 9. A copy of the Form P.2 and the specification of S.A. Patent Application No .
- ☐ 10. Declaration and power of attorney on Form P.3.
- ☐ 11. Request for ante-dating on Form P.4.
- ☐ 12. Request for classification on Form P.9.
- ☒ 13. Form P.2 in duplicate.
- ☐ 14. Other.

| | |
|---|---|
| 74 | ADDRESS FOR SERVICE: SPOOR & FISHER, SANDTON |

Dated: 26 July 2002

SPOOR & FISHER
PATENT ATTORNEYS FOR THE APPLICANT(S)

RECEIVED

REGISTRAR OF PATENTS

SPOOR & FISHER

FORM P.6

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978

# PROVISIONAL SPECIFICATION

(Section 30(1) – Regulation 27)

| OFFICIAL APPLICATION NO. | | LODGING DATE | |
|---|---|---|---|
| 21 | 01 | 22 | 26 JULY 2002 |

FULL NAMES OF APPLICANTS

| | |
|---|---|
| 71 | ~~SCHUMANN SASOL SOUTH AFRICA (PROPRIETARY) LIMITED~~ SASOL SA (PTY) LTD 10.03.03 |

FULL NAMES OF INVENTORS

| | |
|---|---|
| 72 | BENETTI, ALDO<br>FOURIE, JOHAN<br>ZWANE, IVOR MZWANDILE |

TITLE OF INVENTION

| | |
|---|---|
| 54 | FIBRES CONTAINING AN ACTIVE INGREDIENT |

## BACKGROUND OF THE INVENTION

THIS invention relates to a process for incorporating active ingredients into a polymeric fibre, and to a polymeric fibre containing active ingredients.

In many situations, it is desirable to include an active ingredient in a fibrous material. Typical active ingredients include insecticides, anti-bacterials, fungicides and insect repellants. Various methods of including such active ingredients in fibres are known. However, it has proven to be difficult to produce a fibrous material where the active ingredient has a long-lasting effect.

One way of incorporating an active ingredient in a fibrous material or textile is to coat or submerge the material in a liquid composition containing the active ingredient and allow the material to dry. The coating or submerging step is inconvenient, inefficient and expensive and the effect of the active ingredient is not long-lasting as the ingredient is easily removed when the material is washed.

Another way is to heat a fibre (usually a polyester fibre) above its glass transition temperature and then treat the fibre with a liquid containing the active ingredient. While this achieves better incorporation of the active ingredient, the method results in mechanical damage to the fibre during the heating and cooling process.

Japanese patents nos. 8109516 and 4065509 describe methods of making fibres containing active ingredients by melt spinning a mixture of polymer and active ingredients. A problem with these methods is that the step of melt spinning takes place at a high temperature of approximately 160°C and many active ingredients will either be destroyed or vapourised at the high temperature.

It is an object of this invention to provide a new process for incorporating active ingredients into a polymeric fibre, and to produce a polymeric fibre containing a long-lasting active ingredient.

## SUMMARY OF THE INVENTION

According to the first aspect of the invention there is provided a formed polymeric product containing an active ingredient and a wax.

Preferably, the wax has an initial boiling point of at least 300°C at 101.3kPa, and may be selected from Fischer Tropsch (FT), polyethylene or montan waxes.

Advantageously, the product includes a wax and active ingredient in a ratio of 1.5:1 to 0.5:1, usually in a ratio of 1:1.

The product may comprise 90%-98%, by weight, typically 94%-95%, by weight, polymer, 1%-5%, by weight, typically 3%, by weight, wax, and 1-5%, by weight, typically 3%, by weight, active ingredient.

Conveniently, the formed product is an extruded product, preferably a melt-spun extruded product such as a fibre.

A typical active ingredient is an insecticide selected from pyrethroids or phosphates namely a permethrin such as alphacypermethrin.

According to a second aspect of the invention there is provided a method of making a formed product containing an active ingredient, the method including the steps of:

1. preparing a mixture containing a polymer, a wax and an active ingredient; and
2. forming the mixture into the product.

Advantageously, the product is formed by monofilament or multifilament extrusion, preferably monofilament extrusion into a fibre at temperatures of 200°C to 230°C.

## DESCRIPTION OF EMBODIMENTS

This invention relates to a process for incorporating active ingredients into formed polymeric products, particularly polymeric fibres.

The polymeric product consists primarily of polymer materials such as polyvinyl chloride (PVC), polyesters, polypropylene, polyethylene or polyamides etc., or mixtures thereof.

The active ingredients may be insecticides selected from pyrethroids or phosphates, typically a permethrin such as alphacypermethrin. Other active ingredients include anti-bacterials such as triclosane, fungicides such as guazatine and insect repellants such as DEET.

It has surprisingly been found that it is possible to incorporate the abovementioned active ingredients, many of which are volatile, in the formation of a polymeric fibre in a high temperature monofilament extrusion process without degradation of the polymer or active ingredient, by including a wax in the polymer feed to the extruder. The wax should have an initial boiling point which is greater than 300°C at 101.3kPa, and may be selected from Fischer-Trospch, polyethylene and montan waxes. A typical example of a wax is EnHance which is a proprietary Fischer-Tropsch wax of the applicant.

A process according to the invention is carried out mixing a polymer, active ingredient and wax in a tumble-blender to form a feed material for an extruder. The polymer feed will typically comprise 90%-98% by weight, polymer which is suitable to form a fibre (such as a mixture of polypropylene and high density polypropylene), 1%-5%, by weight, typically about 3%, by weight, wax and 1%-5%, by weight, typically 5%, by weight, active ingredient. The mixture is fed to a single-screw extruder with five heating zones (temperatures ranging from 190 to 230°C) through a dye. The filament thus formed goes through a cooling bath (at 40°C) then

through a roller (or "godet") at a suitable speed. After this the fibre is fed through a heating bath (at 96°C) and through another roller set at a suitable speed such that a specific "draw ratio" is achieved. The fibre goes through an oven (90°C) before being fed through a final roller (again at suitable speed) before winding on to a spool. The fibre produced by this process will have a thickness dependant on the die diameter and the speeds of the rollers. It may be knitted to form a fabric containing the active ingredient. In a typical example, a fibre made according to the aforementioned process with the insecticide being alphacypermethrin may be knitted to form a net for protection against mosquitoes. The net produced by such a method may be effective for up to twenty washes performed over an eight week period and still retain its mosquito-repelling properties.

The invention will now be described with reference to the following non-limiting example:

**Example 1**

A solid polymer feed comprising 75 parts polypropylene, 25 parts high density polyethylene, 3 parts EnHance wax having an initial boiling point of 390°C at 101.3kPa and 3 parts alphacypermethrin insecticide was prepared by high speed mixing. This feed was then fed into a single-screw fibre extruder fitted with a die of 0.9mm diameter. The heating zones of the extruder were 190, 200, 210, 214 and 214°C. After extrusion through the die, the filament was fed through a cooling bath at 40°C and the first roller at 14.2m/min. It was then passed through the heating bath at 96°C into the second roller operating at 100.3m/min. draw ratio was 1:7.06. The fibre was then passed through an oven at 90°C before passing through the third roller at 95m/min. This produced a 360 denier fibre. The fibre was knitted to form a net for protection against mosquitoes. The net was tested for efficacy before and after twenty washes performed over a period of eight weeks and was found to retain its mosquito-repelling properties.

Dated this 26TH day of July 2002

Spoor & Fisher

Applicants Patent Attorneys